

## Nanocalorimetry Measurements

### **Facilities**

MSEL researchers have established a state-of-the-art Nanocalorimetry Measurements Facility for determination of thermal properties of materials at sensitivities less than 10 nJ. The nanocalorimeters are chips fabricated at the NIST Advanced Measurement Laboratory Nanofab, chips fabricated in collaboration with the University of Illinois at Urbana-Champaign, and commercially available chips.

Nanocalorimetry experiments may be performed either under ambient conditions, with a controlled gas atmosphere, or in high vacuum. Thin solid films deposited directly on the nanocalorimeter chips and liquid phase and biological samples may be measured. For liquid phases, a droplet generator and precision micropositioners facilitate placement of liquid droplets onto the chips.



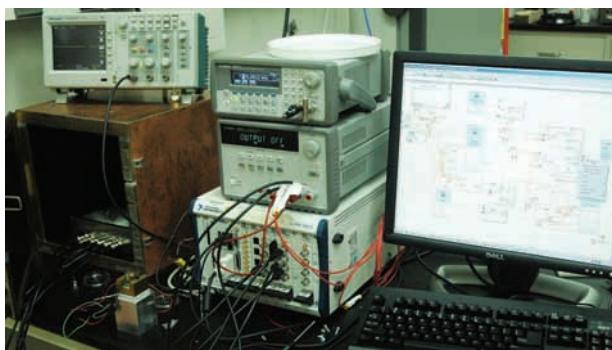
High vacuum nanocalorimeter test system.



A wafer of nanocalorimeter chips

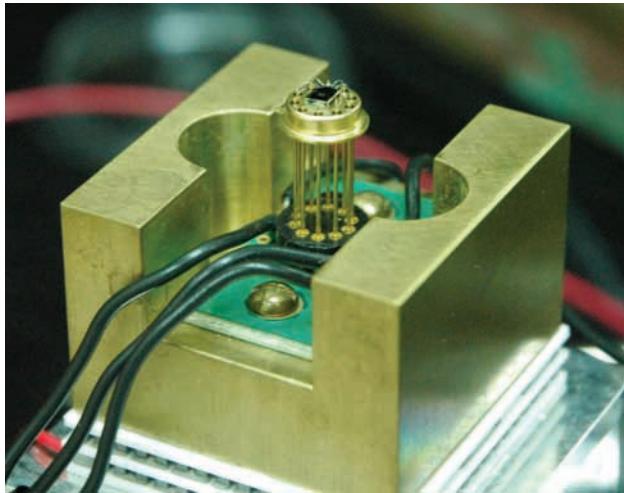
The facility may be used to test and optimize nanocalorimeter chip design and performance, calibrate existing chips, develop new nanocalorimetry calibration methods and standards, and measure the thermal and thermodynamic properties of a wide variety of samples.

The nanocalorimetry facility is composed of two sets of instrumentation. The first set is designed to operate in capacitive discharge mode for rapid heating, but lacks fine pulse shape control. The second set is designed for open loop or closed loop control of the heating and cooling cycles and includes a new 24-bit data acquisition system. Both sets of instrumentation are capable of data acquisition rates of  $2 \times 10^5$  points per second for experiments with heating rates up to 100,000 °C/s.



Nanocalorimeter instrumentation for open or closed loop control of tests.

FACILITIES



A nanocalorimeter chip mounted on a thermoelectric cooler for low temperature testing.



Nanocalorimeter chip calibration system based on an electrical probe station and a state-of-the-art emissivity-correcting pyrometer with a 250  $\mu\text{m}$  diameter working area.

The nanocalorimeter calibration system includes a high vacuum tube furnace capable of 1000 °C, and a probe station-based calibration system coupled with an emissivity corrected pyrometer (250  $\mu\text{m}$  diameter working area) that can measure local temperatures on the nanocalorimeter chips from sub-millimeter regions.

The integrated nature of the facility enables cross-checking between instruments, further increasing accuracy and precision.

### Significance

Advances in many classes of materials depend on accurate measurement of thermodynamic properties. Recently developed NIST nanocalorimeter chips offer a highly sensitive tool for measurements of heat capacity, reaction temperatures, and enthalpy of novel materials. Such measurements of reactions at the nanoscale provide new, detailed insight into nanomaterials, thin film materials, and biological samples. These measurements are essential for commercialization of advanced materials in industries such as packaging and microelectronics, while reducing costs and waste. For example, thermodynamic measurements of silicide formation in microelectronic interconnect materials enables the semiconductor industry to optimize metallization of smaller and smaller transistors and continue to develop faster and more powerful processors and memory chips.

MSEL researchers use the above facility to develop new measurement methods, characterize available measurement systems, and develop calibration materials, methods, and standards to facilitate the expansion of nanocalorimeter measurement science and technology.

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